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(54) MELANGES D'ACIDES CARBOXYLIQUES ET D'AMINES ORGANIQUES POUR LA FLOTTATION DE MINERAI

(54) BLENDS OF CARBOXYLIC ACIDS AND ORGANIC AMINES IN ORE FLOTATION

(57) Alkylamines, alkyldiamines, alkylpolyamines, éther-amines et éther-polyamines, neutralisées par des acides carboxyliques C₃₋₂₄; ces composés possèdent une fluidité améliorée et, dans certains cas, forment des dispersions stables dans l'eau; ils sont efficaces pour la flottation par moussage des impuretés provenant du minerai. Il permettant notamment d'améliorer sensiblement l'élimination à pH élevé des impuretés siliceuses dans le minerai de fer.

(57) Alkyl amines, alkyl diamines, alkyl polyamines, ether amines, and ether polyamines, neutralized with C₃₋₂₄ carboxylic acids have improved fluidity and in some cases form stable dispersions in water, and are effective in froth flotation of impurities from ore. In particular, the removal of siliceous impurities from iron ore at high pH is significantly improved.

ABSTRACT OF THE DISCLOSURE

Alkyl amines, alkyl diamines, alkyl polyamines, ether amines, and ether polyamines, neutralized with C_{3-24} carboxylic acids have improved fluidity and in some cases form stable dispersions in water, and are effective in froth flotation of impurities from ore. In particular, the removal of siliceous impurities from iron ore at high pH is significantly improved.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

- 1. A method of separating impurities from ore containing said impurities, comprising establishing a slurry comprising said ore, water, optional froth flotation additives, and a composition of matter formed by blending:
- (a) an amine component which comprises one or more compounds selected from the group consisting of alkyl amines, alkyl diamines, alkyl polyamines, ether amines, ether polyamines, and mixtures thereof; and
- (b) an acid component selected from the group consisting of linear, branched and cyclic alkenoic and alkanoic carboxylic acids containing 3 to 24 carbon atoms and aromatic carboxylic acids containing 7 to 12 carbon atoms, and mixtures thereof; wherein the amount of the acid component corresponds to about 1 to about 100 mole percent of the nitrogen atoms in said amine component; and

subjecting said slurry to froth flotation, wherein the amount of said composition in said slurry is effective to promote flotation of said impurities from said ore.

2. A method according to Claim 1 wherein said amine component comprises one or more compounds selected from the group consisting of

alkyl amines of the formula R_nNH_{3-n} wherein n is 1, 2 or 3 and each R is independently linear or branched alkyl or alkenyl containing 4 to 24 carbon atoms;

ether amines having the formula R^1 -O- R^2NH_2 wherein R^1 is alkyl containing 4 to 36 carbon atoms and R^2 is alkyl containing 2 to 5 carbon atoms;

ether polyamines having the formula R^3 -O- $(R^4NH)_{1-6}$ - R^5NH_2 wherein R^3 is linear or branched alkyl containing 4 to 36 carbon atoms, R^4 is alkyl containing 2 to 5 carbon atoms, and R^5 is alkyl containing 2 to 5 carbon atoms;

alkylpolyamines having the formula $X-(Alk-N(R^{10}))_{p}-Y$

wherein X is $-NH_2$ or H, p is 2 to 10, each Alk is independently alkyl containing 1 to 6 carbon atoms, each R^{10} is independently -H, alkyl containing 1 to 22 carbon atoms, or alkenyl containing 2 to 22 carbon atoms, and Y is -H, alkyl containing 1 to 22 carbon atoms, or alkenyl containing 2 to 22 carbon atoms, and

alkyl diamines of the formula $(R^6)(R^7)N(CH_2)_2$ $_3N(R^8)(R^9)$ wherein each of R^6 , R^7 , R^8 and R^9 is independently alkyl containing 1 to 30 carbon atoms, and each of R^7 , R^8 and R^9 can independently be hydrogen.

- 3. A method according to Claim 1 wherein said acid component comprises one or more acids containing 6 to 15 carbon atoms.
- 4. A method according to Claim 2 wherein said acid component comprises one or more acids containing 6 to 15 carbon atoms.

- 5. A method according to Claim 2 or 4 wherein R^1 is alkyl containing 6 to 16 carbon atoms; and R^3 is alkyl containing 6 to 16 carbon atoms.
- 6. A method according to any one of Claims 1 to 5 wherein said ore is selected from the group consisting of iron ore, potash ore, phosphate ore and calcite.
- 7. A method according to Claim 6 wherein said ore is iron ore.

BLENDS OF CARBOXYLIC ACIDS AND ORGANIC AMINES IN ORE FLOTATION

The present invention relates to compositions of matter which are blends of amines, particularly alkyl amines, alkyl diamines, alkyl polyamines, ether amines, and ether polyamines, with acids selected from a certain carefully defined class of medium-chain linear or branched carboxylic acids, including any reaction products of such amines and acids that may be present.

The present invention also relates to compositions of matter comprising the aforementioned blends of acids and amines which are useful as surface active agents. The present invention further relates to processes using these novel compositions of matter in applications taking advantage of their superior surface active properties in water, particularly in their use in mineral flotation such as the beneficiation of iron ores. The present invention also relates to the improved physical handling characteristics of these novel compositions especially as compared to corresponding blends with shorter chain carboxylic acids, particularly acetic acid.

Amine compounds such as alkyl amines, alkyldiamines, ether amines, ether diamines, and polyamines, exhibit surface active properties particularly in aqueous media which make such compounds useful in a host of applications.

One such application is the beneficiation of ores by froth flotation, particularly the removal of

silica from iron ores or phosphate ores, the recovery of potash from sylvite, and the purification of calcite.

In the froth flotation of silica from e.g. iron ore hydrophobic amine compounds are added to a pulp of finely ground ore causing the silica particles to become hydrophobic by selectively adsorbing on them. The hydrophobic silica particles are then removed from the slurry by attachment to air bubbles rising through the pulp. The relatively more hydrophilic iron ore particles are unaffected by the air bubbles and remain in the pulp.

The hydrophobic nature of the amine is critical to the froth flotation process, but it causes problems in obtaining a uniform mixture of amine in the ore pulp due to its immiscibility in water. The small amount of amine used (0.02 - 2 pounds per ton of ore treated) requires a uniform dilute dispersion of amine in the pulp in order to obtain optimum coverage of the silica particles. Two methods are generally used to effectively deliver the amine to the silica surface.

One method used is to disperse the amine in water and then meter the dispersion into the froth flotation slurry. The use of a dilute amine dispersion improves amine consumption and eases the metering problems associated with the small amount of amine used. It is convenient to store the dispersion for 8 to 12 hours while metering it into the flotation cell. A storage stable dispersion is one that remains dispersed for 12 hours without agitation. The hydrophobic amine products used in silica flotation will not form storage

stable dispersions in water unless a dispersant is used. The choice of dispersing aids is limited due to the need to disperse the amine without forming an emulsion or interfering with the attachment of amine to the silica surface. The dispersing aid generally used heretofore has been acetic acid either directly in the dispersion water, or as amine products partially neutralized or blended with acetic acid. The need to handle a corrosive acid in the flotation plant environment is a drawback to this method. Directly adding the acid to the amine to form an amine salt as a dispersing aid at the point of manufacture solves the problem of acid handling. However, for lower molecular weight amines, such as C₈ - C₁₀ ether primary amines, the by-products of the amine and acetic acid reaction can be very odoriferous and unpleasant to work with.

Also, acetic acid-neutralized ether primary amines can form very stable foams if air is mixed in (even inadvertently) such as can occur during packaging, transporting and/or unloading. Further, the acetic acid-neutralized amines and diamines are higher melting and more viscous which makes handling more difficult and, in some cases, leads to the formation of amides at the temperatures necessary to maintain liquidity. In some cases, amine-acetate dispersions become nonuniform with an oily layer or creamy layer at the top in 24-48 hours, even in as little as 8 hours, and thus cannot be viewed as being long term stable.

The second method meters the amine directly into the flotation slurry relying on the shear generated

by the agitated slurry to disperse the amine. While this method solves the potential odor problem, it requires accurate metering pumps to deliver the proper amount of amine and relies on perfect mixing in the flotation cell in order to obtain satisfactory amine usage levels and selectivity.

Amines lose their surfactant nature and their utility as flotation reagents at pH above about 10.5 and act as oils. Flotation of siliceous material from iron ore carried out at these high pH levels is marked by higher consumption and lower yield due to the lack of surface active amine in the system. Flotation of fine grained hematite ores is generally carried out at pH 10.5 - 11 and would benefit by having a more surface active amine reagent.

Accordingly, there is still a need for compositions of matter and particularly aqueous systems containing amines of the aforementioned types whose efficacy, as may be influenced by their dispersability, is enhanced by suitable acidic agents without the generation of undesirable odors, or by-products, and without suffering loss of desired properties of the amine components. Also, there is a need for amine based flotation reagents that do not lose effectiveness when used at pH 10 or above.

The present invention satisfies these objects and avoids the drawbacks of prior approaches, and also provides the additional advantages enumerated herein.

The present invention relates to compositions of matter comprising a blend of (a) an amine component

which is one or more compounds selected from the group consisting of alkyl amines, alkyl diamines, alkyl polyamines, ether amines, ether polyamines, and mixtures thereof; and (b) an acid component selected from the group consisting of linear, branched and cyclic, alkenoic, alkanoic and aromatic carboxylic acids containing 3 to 24 and preferably 6 to 13 carbon atoms; wherein the amount of the acid component corresponds to about 1 to 100 mole percent, and preferably about 3 to about 70 mole percent, of the number of nitrogen atoms of the amine component.

In addition to the above-noted blends, the present invention is also directed to products which are dispersions in water of any of such blends. Such dispersions, especially when the acid component comprises linear acids, form readily and remain stable, and exhibit the other advantages described herein.

Thus, the present invention is further directed to a method of forming an aqueous homogeneous storage-stable dispersion of one or more of said amines (a) as defined hereinabove, the method comprising dispersing said one or more amines with one or more of said acids (b) as defined hereinabove and adding the resulting dispersed blend to water while mixing with mild aqitation.

Still even further, the present invention is directed to a method of removing impurities from ore, such as iron ore, potash ore, phosphate ore, or calcite, comprising subjecting a slurry of said ore in water to froth flotation, wherein the water contains dispersed

therein a composition of matter as set forth herein, in an amount of said composition in said slurry effective to promote flotation of said impurities.

Amines with which this invention is useful include alkyl amines, alkyl diamines, alkyl polyamines, ether amines, and ether polyamines. Said amines can be primary, secondary or tertiary amines.

Alkyl amines with which the present invention is useful include those of the formula R_nNH_{3-n} wherein R is linear or branched alkyl or alkenyl (i.e. containing a carbon-carbon double bond) of 4 to 24 carbon atoms, and n is 1, 2 or 3.

Ether amines with which this invention is useful include those having the formula R¹-O-R²NH₂, wherein R¹ is branched or linear alkyl or alkenyl containing 4 to 36 carbon atoms and R² is alkyl containing 2 to 5 carbon atoms. Preferably, R¹ is alkyl containing 6 to 15 carbon atoms, and more preferably 8 to 13 carbon atoms and yet more preferably 8 to 10 carbon atoms. R² is preferably alkyl containing 3 carbon atoms, as would result from Michael addition reactions using acrylonitrile.

Ether diamines or ether polyamines with which the present invention is useful include those having the formula R³-O-(R⁴NH)₁₋₆-R⁵NH₂ wherein R³ is linear or branched alkyl or alkenyl containing 4 to 36 carbon atoms, each R⁴ is independently alkyl containing 2 to 5 carbon atoms, and each R⁵ is alkyl containing 2 to 5 carbon atoms. Preferably, R³ is alkyl containing 6 to 15 carbon atoms, and yet more preferably 8 to 12 carbon

atoms. R^4 is preferably alkyl containing 2 to 3 carbon atoms, and R^5 is preferably alkyl containing 2 to 3 carbon atoms.

Alkyl polyamines useful in the present invention include those having the formula X-(Alk-N(R¹⁰)) $_p$ -Y wherein X is -NH $_2$ or -H, p is 2 to 10, each Alk group is independently alkylene containing 1 to 6 carbon atoms, each R¹⁰ is independently -H or alkyl containing 1 to 22 carbon atoms, and Y is -H, alkyl containing 1 to 22 carbon atoms, or alkenyl containing 2 to 22 carbon atoms. In preferred polyamines of this formula, p is 3 to 5, each Alk is ethylene or 1- or 2-propylene, each R¹⁰ is -H, methyl or ethyl, and Y is -H or alkyl containing 1 to 6 carbon atoms.

Preferred alkyl diamines useful in the present invention include those having the formula $(R^6)(R^7)N(CH_2)_{2-3}N(R^8)(R^9)$ wherein each of R^6 , R^7 , R^8 and R^9 is alkyl containing 1 to 30 carbon atoms, and preferably alkyl containing 4 to 20 carbon atoms. Also, the groups R^{6-9} can be linear, branched, cyclic, or aromatic (especially phenyl). Moreover, any of R^7 , R^8 and/or R^9 can be hydrogen.

The amines, diamines, and polyamines of any of the foregoing formulas can readily be formulated in accordance with known synthetic techniques. Indeed, many of the amines of the foregoing structures are commercially available.

In all of the foregoing formulas, any or all alkyl groups can be linear or branched, and saturated or unsaturated.

Acids useful in the present invention include linear, branched and cyclic alkanoic and alkenoic carboxylic acids containing 3 to 24 carbon atoms and preferably 6 to 13 carbon atoms. Useful acids also include aromatic carboxylic acids containing 7 to 12 carbon atoms, such as benzoic acid. This acid component can comprise one acid but preferably comprises a mixture of two or more such acids. Acids corresponding to this description, as well as mixtures thereof, are readily synthesized and many are also commercially available. Preferred examples include the mixtures of branched-chain carboxylic acids marketed by the Exxon Corporation under the trade names "NEO 908" and "NEO 913", or the straight-chain carboxylic acid blend marketed by Witco Corp. under the name "Industrene 365".

The relative amounts of the amine component and the acid component can range widely, and accordingly can be tailored to optimization of the particular properties and intended applications of the resulting composition of matter. In general, the amount of acid corresponds to at least about 2 percent based on the total number of nitrogen atoms present in the amine component. More preferably, the amount of acid can correspond to at least about 10%, up to about 50% or even higher, e.g., up to 80% to 100% on that basis. Thus, it should be understood that the term "neutralization" as used herein is not limited to situations of complete neutralization of all amine sites with acid, but extends also to blends wherein the amount

of acid is only sufficient to partially neutralize the amine. Even partial neutralization provides improved performance as described herein.

When neutralization is carried out to improve the dispersibility in water of the amine, the extent of neutralization required depends strongly on the molecular weight of the amine. Short chain amines require very little neutralization to achieve proper dispersion whereas longer chain amines require higher neutralization to disperse as desired in cold water. In some cases neutralization with middle chain-length acids does not yield stable dispersions, but nonetheless, surprisingly, improves ore flotation.

The products useful in this invention are termed "blends", which can be formed by simply combining the acid or acids, and the amine or amines, in the amounts indicated, in any suitable manner that forms a uniform mixture thereof. The term "blend" used herein denotes the resulting mixture and encompasses not only compositions composed entirely of discrete acids and amines, but also compositions containing one or more reaction products of acid and amine, typically acid salts.

The composition of matter is preferably provided as 100% active but may be dissolved or dispersed or emulsified in appropriate organic solvents water, or aqueous solution.

It has been determined that the compositions prepared in accordance with the present invention exhibit enhanced surface active performance in selected

applications, coupled with ease of handling as a neat liquid and freedom from by-products. Especially advantageous is the freedom of these compositions from any by-products causing an unpleasant odor. Heating blends of an amine and short chain carboxylic acid can cause the formation of by-products that have a very unpleasant odor. The formation of by-products decreases the activity of the amine and acid in the blend and thus leads to a shorter storage life.

Also, the compositions retain desired liquidity at temperatures lower than has been the case with prior products that needed to be heated to remain liquid.

The present invention is illustrated in the following examples.

EXAMPLE 1

Three amines, coco fatty primary amine (commercially available as Adogen 160D, Witco Chemical), n-dodecyl ether primary amine, and isododecyl ether amine, were 100% neutralized with three acids (acetic acid, C_8 - C_{10} linear carboxylic acid and branched C_9 - C_{13} carboxylic acid). The samples were placed in an 80°C oven for two weeks. Total amine value (or TAV --a measure of the active amine in the sample) was taken on each sample before and during oven storage to determine the stability.

The results are listed in Table 1. They show that the present invention exhibits greatly reduced loss of the amine to by-products, even over time, which means that the present invention makes a higher proportion of the amine available for its desired function.

Table 1
TAV of amine acid blends stored at 80°c

amine/acid	Start	1 day	7 day	14 day	% drop
coco/acetic	218	205	138	102	53
isododecyl/ acetic	171	162	103	65	62
n-dodecyl acetic	181	163	99	67	63
coco/C ₈₋₁₀	159	148	111	80	50
isododecyl/ C ₈₋₁₀	135	123	84	54	60
n-dodecyl/ C ₈₋₁₀	138	130	79	54	61
coco/C ₉₋₁₃	158	162	161	154	3
isododecyl/ C,.13	138	140	142	133	4
n-dodecyl/ C ₉₋₁₃	143	142	143	140	2

EXAMPLE 2

The viscosity vs. temperature properties of the samples tested in Example 1 were determined. Each sample viscosity was measured in the Brookfield viscometer small sample cell that was temperature controlled by a programmable hot bath. The temperature was raised to 45°C and slowly lowered to 10°C. Viscosity measurements were made at 2 minute intervals. The linear amines and salts were marked by a sharp solidification point (arbitrarily set at cP of 200,000) while the branched amines and salts generally remained below the pour point

(arbitrarily set at cP 10,000). The viscosity data is summarized below:

Amine Salt	Solid	Pour
Cocoamine ("Adogen 160")	10°C	15°C
Acetate	30	45
C ₉₋₁₃	<10	25
C ₈₋₁₀	20	25
Isododecyl ether amine	<10	<10
Acetate	<10	<10
C ₉₋₁₃ .	<10	20
C ₈₋₁₀	<10	<10
n-dodecyl ether amine	<10	<10
Acetate	45	55
C ₉₋₁₃	<10	15
C ₈₋₁₀	10	15

Typically acetic acid is used as the dispersant for amine in water dispersions. Many of the longer chain carboxylic acids can also act as dispersants for amine in water systems. This aspect of the invention is illustrated in the following example.

EXAMPLE 3

An ether amine corresponding to the general formula $(C_8-C_{10} \text{ alkyl})-O-CH_2-CH_2-CH_2-NH_2$ (commercially available from Witco Corporation as "Arosurf MG 98") was neutralized 50% with various chain length carboxylic acids. The neutralized MG 98 was used to make dispersions of 6% amine in room temperature tap water. The dispersions were allowed to sit undisturbed to determine the stability of the dispersion formed. A dispersion was considered stable if there was no evidence of separation in 2 hours. The results are indicated as stable or unstable dispersions:

Acid	Dispersion
Acetic	Stable
Propionic	Stable
Butyric	Unstable
Hexanoic	Stable
2-Ethyl hexanoic	Unstable
2-Methyl valeric	Stable
C ₈ -C ₁₀ Acid	Stable
Tall oil	Unstable
Neo 913	Unstable

In particular, compositions in accordance with the present invention have been found to be unexpectedly highly beneficial in the froth flotation of impurities, particularly siliceous impurities, from ore such as iron ore, potash ore, phosphate ore, and calcite. Such impurities generally comprise silica, inorganic silicates, and mixed silicates such as aluminosilicates. This advantageous application will now be described in further detail.

Most of the particulars of the froth flotation are carried out in accordance with conventional, wellknown apparatus and conditions familiar to those of ordinary skill in this art. Finely divided ore is slurried in water (at a solids content on the order of 25 wt.% to 50 wt.%) in a flotation cell equipped to inject air (or gas) into the slurry at a rate sufficient to form a froth of bubbles at the top of the cell. froth is removed by skimmers, paddles or other conventional techniques. Additives can be added, and usually are added, to the slurry to improve the separation provided by the flotation cell. additives can include frothers, which promote the formation of the froth, activators, promoters, and collectors, which improve the separation by increasing the affinity of the floated material for the froth, or, optionally, depressants for decreasing the affinity for the froth of the material which is desired to remain unfloated in the flotation cell, and pH regulators for adjusting the pH of the ore slurry to the desired level to obtain optimum utility from the additives mentioned.

In iron ore processing, because of the relative weights involved and because of the details of the surface chemistry, it is desired to float the impurities in the froth, leaving the ore thus beneficiated in the pulp in the slurry.

Addition of a composition of matter comprising an amine component and an acid component, in accordance with the description herein, when added to a slurry of hematite ore at pH 10.5 containing siliceous impurities, is effective in promoting flotation of the siliceous impurity from the iron ore. Typical amounts of this promoter to add range from about 0.001 wt.% to about 0.06 wt.%, and preferably 0.0025 wt.% to about 0.025 wt.% of the ore. This advantageous result is described further in the following specific examples.

EXAMPLE 4

An ether amine corresponding to the general formula (C₈-C₁₀ alkyl)-O-CH₂CH₂CH₂NH₂ (commercially available from Witco Corporation as "Arosurf MG 98") was neutralized 40% with a commercially available carboxylic acid product comprised predominantly of branched carboxylic acids with 9 to 13 carbon atoms, commercially available from Exxon Corp. as "NEO 913". This amineacid blend was compared, for effectiveness in removing siliceous impurities from magnetite iron ore concentrate, to unneutralized "Arosurf MG-98". One test series was carried out with the iron ore slurry at pH 8.5 and another with the slurry at pH 11.0. In both cases the slurry was 35 wt.% iron ore, and equal amounts of amine sample were used.

The following table sets forth the iron recovery in the froth flotation step, as a function of the iron grade of the magnetite concentrate produced, for the flotation runs made with unneutralized "Arosurf MG-98" and with "Arosurf MG-98" blended 40% with "NEO 913". The results for pH 8.5 and pH 11.0 are shown in Table 2.

TABLE 2

Comparison of Magnetite Iron Ore Flotation with AROSURF MG-98, and AROSURF MG-98 Neutralized 40% with NEO 913

$At_pH=8.5$

With MG-98

Iron Recovery (%)
59.8
68.9
79.3
86.0

With MG-98/40% Neutralized

Iron Grade (%)	Iron Recovery (%)
68.3	71.2
68.2	75.1
68.0	82.7
67.5	90.0

At pH=11.0

With MG-98

Iron Grade(%)	Iron Recovery (%)
67.7	. 80.5
67.67	82.0
67.43	86.5
67.01	91.0

With MG-98/40% Neutralized

Iron Grade (%)	Iron Recovery (%)
67.7	81.0
67.52	84.8
67.2	88.7
66.74	92.9

The results of flotation at pH 8.5 are expected due to the fact that the 40% neutralized material is only 77% by weight amine and should be less effective than 100% active amine. The especially surprising result is the quality of the neutralized reagent when flotation was done at pH 11.0. At this pH the amine in the neutralized product is being utilized more efficiently than the unneutralized amine to obtain the equivalent results noted.

EXAMPLE 5

"Arosurf MG-98" and a sample of "Arosurf MG-98" that was 30% neutralized with a mixture of linear C_{B} - C_{10} carboxylic acids were compared for flotation effectiveness on a nonmagnetic ore. The slurry that was tested comprised about 35 wt.% hematite iron ore and had a pH of 10.5. A comparison was made of the effect of the ether amine compared to an equal amount of ether amine that had been neutralized with the carboxylic acid mixture. The amount of amine employed was about 0.02 wt.% of the amount of iron ore in the slurry. following Table 3 sets forth the iron recovery in the froth flotation step, as a function of the iron grade of the hematite material left in the flotation bowl, for the flotation runs made with the unneutralized ether amine and with the same ether amine neutralized with the carboxylic acid mixture.

TABLE 3

Hematite Iron Ore, Tilden, Michigan

Unneutralized MG-98 Ether amine

Iron Grade(%)	<pre>Iron Recovery(%)</pre>
61.3	53.3
61.2	64.8
60.9	69.2
59.3	75.5

MG-98 Neutralized 30% with C_8-C_{10} Acids

<pre>Iron Grade(%)</pre>	<pre>Iron Recovery(%)</pre>
61.5	58.8
61.4	69.2
61.1	73.3
60.0	77.7

These results, demonstrate that for any given grade of iron ore the recovery of the iron ore is superior when the froth flotation is carried out with partially neutralized ether amine as described hereinabove in this application.